MECHANICAL BEHAVIOUR OF BAGASSE FIBER EPOXY COMPOSITES AT LIQUID NTROGEN TMPERATURE

A PROJECT REPORT SUBMITTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS

FOR

THE DEGREE IN

Bachelor of Technology

In

Mechanical Engineering

By

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Under the Guidance

Of

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Rourkela
2009

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CERTIFICATE

This is to certify that the thesis entitled, “MECHANICAL BEHAVIOUR OF BAGASSE FIBER EPOXY COMPOSITES AT LIQUID NITROGEN TEMPERATURE” submitted by Sri DEBABRATA DEBNATH in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Mechanical Engineering at the NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

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ACKNOWLEDGEMENTS

It is with a feeling of great pleasure that I would like to articulate my most sincere heartfelt gratitude to Prof. S.K. Acharya, Department. of Mechanical Engineering, NIT Rourkela for suggesting the topic for my thesis report and for his ready and able guidance through out the course of my preparing the report. I am greatly indebted to him for his constructive suggestions and criticism from time to time during the course of progress of my work.

I express my sincere thanks to Prof. R.K. Sahoo, Head of the Department of Mechanical Engineering, NIT, Rourkela for providing me the necessary facilities in the department and for his valuable guidance, constant encouragement and kind help at different stages for the execution of this dissertation work.

I express my sincere gratitude to Prof. K.P.Maity, Co-ordinator of M.E. course for his timely help during the course of work.

I would also express my thanks to Mr. Bishwanath Mukherjee of Department. Of Mechanical Engineering, for helping me in understanding the whole procedure in Liquid nitrogen production and maintenance.

I am thankful to Mrs. Punyapriya Mishra for her co-operation in the experimental work.

I would also like to thank my parents without whose inspirations and blessings I would not have been able to reach this far.

Last but not the least I will be grateful to all my friends who have stood by me when I needed them to during the course of the project.

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ABSTRACT

India is a country which lives still in its villages and the farm products constitute the separate joints and bones of the backbone of the country's economy out of which natural fiber has been an important and profitable product since the beginning of business. In recent years the natural fiber epoxy composites have attracted substantial importance as a potential structural material. The mechanical properties of several types of epoxy systems are designed based on the chemical structure, network structure and morphology aiming at cryogenic applications such as cryogenic wind tunnels, cryogenic transport vessels, support structures in space shuttles and rockets. In these applications they are often under cyclic loading. The attractive features of natural fibers like jute, sisal, coir and banana have been their low cost, light weights, high specific modulus, renew ability and biodegradability. Natural fibers are lignocellulosic in nature. These composites are gaining importance due to their non-carcinogenic and bio-degradable nature. The natural fiber composites can be very cost effective material especially for building and construction industry. However in many instances residues from traditional crops such as rice husk or sugarcane bagasse or from the usual processing operations of timber industries do not meet the requisites of being long fibers. Bagasse contains about 40% cellulose, 30% hemicellulose, and 15% lignin. The present use of bagasse is mainly as a fuel in the sugar cane mill furnaces. It is felt that the value of this agricultural residue can be upgraded by bonding with resin to produce composites suitable for building materials.

Keeping this in view the present work has been undertaken to develop a polymer matrix composite (epoxy resin) using bagasse fiber as reinforcement with volume fractions 10,15 & 20% and to study its mechanical properties at cryogenic temperature.

In the next part of this project the main emphasis was laid on the experimental work relating to the mechanical behaviour of this composite at liquid nitrogen temperature.

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CHAPTER 1
INTRODUCTION

India endowed with an abundant availability of natural fiber such as Jute, Coir, Sisal, Pineapple, Ramie, Bamboo, Banana etc. has focused on the development of natural fiber composites primarily to explore value-added application avenues. Such natural fiber composites are well suited as wood substitutes in the housing and construction sector. The development of natural fiber composites in India is based on two pronged strategy of preventing depletion of forest resources as well as ensuring good economic returns for the cultivation of natural fibers.

The developments in composite material after meeting the challenges of aerospace sector have cascaded down for catering to domestic and industrial applications. Composites, the wonder material with light-weight; high strength-to-weight ratio and stiffness properties have come a long way in replacing the conventional materials like metals, wood etc. The material scientists all over the world focused their attention on natural composites reinforced with Jute, Sisal, Coir, Pineapple etc. primarily to cut down the cost of raw materials.

1.1 WHY A COMPOSITE?

Over the last thirty years composite materials, plastics and ceramics have been the dominant emerging materials. The volume and number of applications of composite materials have grown steadily, penetrating and conquering new markets relentlessly. Modern composite materials constitute a significant proportion of the engineered materials market ranging from everyday products to sophisticated niche applications. While composites have already proven their worth as weight-saving materials, the current challenge is to make them cost effective. The efforts to produce economically attractive composite components have resulted in several innovative manufacturing techniques currently being used in the composites industry. It is obvious, especially for composites, that the improvement in manufacturing

technology alone is not enough to overcome the cost hurdle. It is essential that there be an integrated effort in design, material, process, tooling, quality assurance, manufacturing, and even program management for composites to become competitive with metals.

1.2 DEFINITION OF COMPOSITE

The most widely used meaning is the following one, which has been stated by Jartiz [1] “Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form”.

The weakness of this definition resided in the fact that it allows one to classify among the composites any mixture of materials without indicating either its specificity or the laws which should given it which distinguishes it from other very banal, meaningless mixtures.

Kelly [2] very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength to resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Beghezan [3] defines as “The composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their short comings”, in order to obtain improved materials.

Van Suchetclan [4] explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

1.3 CHARACTERISTICS OF THE COMPOSITES

Composites consist of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous phase and is called the 'reinforcement' or 'reinforcing material', whereas the continuous phase is termed as the ' matrix'.

Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and size distribution) influences the properties of the composite to a great extent. The concentration distribution and orientation of the reinforcement also affect the properties.

The shape of the discontinuous phase (which may by spherical, cylindrical, or rectangular cross-sanctioned prisms or platelets), the size and size distribution (which controls the texture of the material) and volume fraction determine the interfacial area, which plays an important role in determining the extent of the interaction between the reinforcement and the matrix.

Concentration, usually measured as volume or weight fraction, determines the contribution of a single constituent to the overall properties of the composites. It is not only the single most important parameter influencing the properties of the composites, but also an easily controllable manufacturing variable used to alter its properties.

1.4 COMPONENTS OF A COMPOSITE MATERIAL

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the 'matrix'), and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

1.4.1 MATRIX

Many materials when they are in a fibrous form exhibit very good strength property but to achieve these properties the fibres should be bonded by a suitable matrix. The matrix isolates the fibres from one another in order to prevent abrasion and formation of new surface flaws and acts as a bridge to hold the fibres in place. A good matrix should possess ability to deform easily under applied load, transfer the load onto the fibres and evenly distributive stress concentration.

1.4.2 REINFORCEMENT

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system. All of the different fibers used in composites have different properties and so affect the properties of the composite in different ways. For most of the applications, the fibers need to be arranged into some form of sheet, known as a fabric, to make handling possible. Different ways for assembling fibers into sheets and the variety of fiber orientations possible to achieve different characteristics.

1.4.3 INTERFACE

It has characteristics that are not depicted by any of the component in isolation. The interface is a bounding surface or zone where a discontinuity occurs, whether physical, mechanical, chemical etc. The matrix material must “wet” the fibre. Coupling agents are frequently used to improve wettability. Well “wetted” fibres increase the interface surfaces area. To obtain desirable properties in a composite, the applied load should be effectively transferred from the matrix to the fibres via the interface. This means that the interface must be large and exhibit strong adhesion between fibres and matrix. Failure at the interface (called debonding) may or may not be desirable.

1.5 CLASSIFICATION:

Composite materials can be classified into many categories depending on the type of matrix material, reinforcing material type etc.

According to the type of **matrix material** they can be classified as follows:

- Metal matrix type composites: MMC are composed of a metallic matrix(Al ,Mg, Fe, Co, Cu)
- Ceramic matrix composites: CMC is a material consisting of a ceramic combined with a ceramic dispersed phase.
- Polymer matrix material: PMC are composed of a matrix from thermosetting (unsaturated polyester, epoxy) or thermoplastic (nylon, polystyrene) and embedded glass carbon, steel or Kerler fibers (dispersed phase). Some of the major advantages and limitations of resin matrices are shown in Table

Advantages and limitations of polymeric matrix materials

Advantages	Limitations
Low densities	Low transverse strength
Good corrosion resistance	Low operational temperature limits
Low thermal conductivities	
Low electrical conductivities	
Translucence	
Aesthetic Color effects	

Generally speaking, the resinous binders (polymer matrices) are selected on the basis of adhesive strength, fatigue resistance, heat resistance, chemical and moisture resistance etc. The resin must have mechanical strength commensurate with that of the reinforcement. It must be easy to use in the fabrication process selected and also stand up to the service conditions.

According to the type of reinforcing material type they can be classified into the following categories:

- **Particle composites**—particle reinforced composites consist of a matrix reinforced by a dispersed phase in the form of particles. It can beeither of random orientation or preferred orientation.

- **Fibrous composites**—Short fiber: they consist of a matrix reinforced by a dispersed phase in the form of discontinuous fibers either of random or preferred orientations.
- **Long fiber**- they consist of a matrix reinforced by a dispersed phase in the form of continuous fibers. They can be either unidirectional or bidirectional.
- **Laminate composites**—when a fiber reinforced composite consists of several layers with different fiber orientations ,it is called multilayer composite.

Apart from that the two broad classes of composites are (1) Particulate composites and (2) Fibrous composites.

1.5.1 Particulate Composites

As the name itself indicates, the reinforcement is of particle nature (platelets are also included in this class). It may be spherical, cubic, tetragonal, a platelet, or of other regular orirregular shape, but it is approximately equiaxed. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composite to a limited extent. Particle fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, improve performance at elevated temperatures, reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage.

1.5.2 Fibrous composites

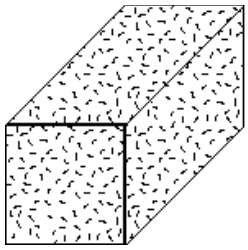
A fiber is characterized by its length being much greater compared to its cross-sectional dimensions. The dimensions of the reinforcement determine its capability of contributing its properties to the composite. Fibers are very effective in improving the fracture resistance of the matrix since a reinforcement having a long dimension discourages the growth of incipient cracks normal to the reinforcement that might other wise lead to failure, particularly with brittle matrices

Man-made filaments or fibers of non polymeric materials exhibit much higher strength along their length since large flaws, which may be present in the bulk material, are minimized because of the small cross-sectional dimensions of the

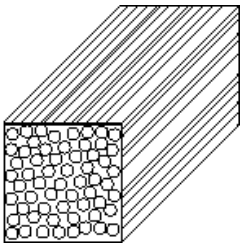
fibre. In the case of polymeric materials, orientation of the molecular structure is responsible for high strength and stiffness.

Fibers, because of their small cross- sectional dimensions, are not directly usable in engineering applications. They are, therefore, embedded in matrix materials to form fibrous composites. The matrix serves to bind the fibers together, transfer loads to the fibers, and protect them against environmental attack and damage due to handling. In discontinuous fibre reinforced composites, the load transfer function of the matrix is more critical than in continuous fibre composites.

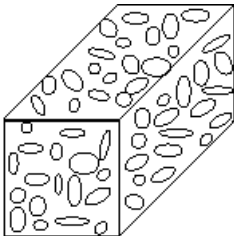
Fig 1.1: Different types of composite materials



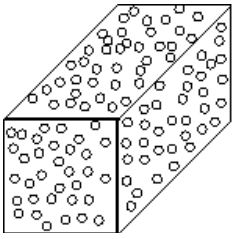
Short fiber composites



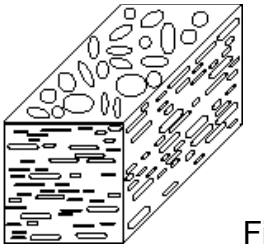
Long fiber composites



Flake composites



Particulate composites



Filler composites

CHAPTER 2
LITERATURE SURVEY

In recent years, the natural fibers have attracted substantial importance as a potential structural material. The attractive plus point of natural fiber si n terms of industrial usage has made its availability more demanding

Keeping this in view the present work has been undertaken to develop a polymer matrix composite (epoxy resin) using bagasse fiber as reinforcement and to study its mechanical properties and performance. The composites are prepared with different volume fraction of bagasse fiber.

Usually the fiber reinforcement is done to obtain high strength and high modulus. Hence it is necessary for the fiber to possess higher modulus than the matrix material. So the load is transferrred to the fiber from the matrix more effectively.

Fiber reinforced composites are popularly being used in many industrial applications because of their high specific strength & stiffness. Due to their excellent structural performance these composites are gaining potential also in tribological applications.

The physical properties of natural fibers are mainly determined by the chemical & physical composition, such as structure of fibers' cellulose content, angle of fibrils, cross section and by the degree of polymerization. Only a few characteristics values but especially the specific mechanical properties can reach the compensable values of traditional fibers. The application of natural fibers as reinforcing materials in composite materials require as just for glass fiber reinforced composites, a strong adhesion between the fiber and the matrix regardless of whether a traditional polymer(thermoplastic or thermosetting) matrix, a biodegradable polymer matrix or cement is used. The mechanical and other physical properties of the composites are generally dependent on the fiber content, which also determines the possible amount of coupling agents in the composite.

An important property of natural fibers to be used as reinforcements is their availability in large quantities. For several more technical oriented applications, the fibers have to be specially prepared or modified regarding

-homogeneity of the fiber's properties

-degrees of elementarization and degumming

-degree of polymerization and crystallization.

-good adhesion between fiber and matrix

-moisture repellent properties

-Flame retardant properties

Nowadays natural fibers are very fast replacing the traditional manmade fibers as reinforcements they have several advantages over manmade fibers.

-plant fibers are renewable and their availability is more or less unlimited.

-when natural fiber composite were subjected to at the end of their life cycle, to a combustion process or landfill the amount of CO2 released of the fibers is neutral with respect to their assimilated amount during their growth.

The abrasive nature is lower which leads to advantages regarding technical material recycling

-natural fiber reinforced plastics by using biodegradable polymers as matrix are the most environment friendly materials.

The main chemical constituents of bagasse are hemi cellulose and lignin. Hemi cellulose and cellulose are present in the form of holocellulose in bagasse, which contributes more than 70 % of the total chemical constituent present in bagasse. Another important chemical constituent present in bagasse is lignin. Lignin acts as a binder for the cellulose fibers and also behaves as an energy storage system.

Usamani etal **[3]** describes the evolution of five water soluble phenolic resin as binders at 5 percent concentration, for oriented and random reinforced bagasse composite. They tried to determine the amount of resin retained during processing when these phenolics were precipitated on to bagasse fiber.

Monteiro SN. Rodriquez etal. **[6]** tries to use the sugar cane bagasse waste as reinforcement to polymeric resins for fabrication of low cost composites. They

reported that composites with homogeneous microstructures could be fabricated and mechanical properties similar to wooden agglomerates can be achieved.

A.Vazquez, V.A.Dominguez etal. **[9]** in their work reported the processing and properties of bagasse fiber-polypropylene composites. Four different chemical treatments were done on fiber to improve interface adhesion with the thermoplastic matrix namely isocyanate, acrylic acid, mercerization and washing with alkaline solution. Their result shows that the best results were obtained on materials with treated fibers.

Hassan etal. **[16,17]** have converted the bagasse into a thermo formable material through esterification of the fiber matrix. The dimensional stability and mechanical properties of the composites prepared from the esterified fibers were reported in this work.

Paiva etal. **[18]** analyzed the impact strength and hardness of sugarcane bagasse-resol composites and showed that impact strength increased and hardness diminished as the fiber volume fraction increased.

Jane M. F.Paiva, E.Frollini **[19]** used short sugar cane fibers as reinforcement to obtain fiber reinforce composites. Lignin extracted from sugarcane bagasse was used as a partial substitute of phenol (40w/w) in resole phenolic matrices. They characterized the composite by mechanical tests such as impact, DMTA and hardness tests. The results as a whole showed that it is feasible to replace part of phenol by lignin in phenolic matrices without loss of properties.

M.V.desousa etal. **[12]** studied the effect of three processing parameters on the flexural mechanical behaviour of chopped bagasse poly-ester composite. The parameters evaluated were: the size of the chopped material, the pretreatment derived from the previous processing of the bagasse material on mills and the molding pressure. The results obtained by them enable the selection of the best combination of bagasse origin, size and molding pressure.

Shinichi Shibata, Yong Cao, and Isao Fukumoto **[21]** in their work investigated experimentally the flexural modulus of the press molding composites made from bagasse fiber and biodegradable resin. They have also numerically predicted flexural modulus by using Cox's model. They conclude that up to 65% volume

fraction of reinforcement flexural modulus increases. Decrease in the flexural modulus was found below 3 mm at the fiber length in the experimental and same trend was shown in the numerical prediction.

Mechanical properties of biodegradable composites reinforced with bagasse fiber with alkali treatment have been reported by Cao etal. **[8].** Approximately 13 % improvement in tensile strength, 14 % in flexural strength and 30% in impact strength has been reported.

BC Ray Department of Metallurgical and Materials Engineering, National Institute of Technology, Rourkela **[20]** used 3-point flexural test to qualitatively assess such effects for 55, 60 and 65 weight percentages of E-glass fibers reinforced epoxy composites during cryogenic and after thawing conditions. The specimens were tested at a range of 0.5 mm/min to 500 mm/min crosshead speed to evaluate the sensitivity of mechanical response during loading at ambient and sub-ambient (-80°C temperature). These shear strength values are compared with the testing data of as-cured samples.

After reviewing the existing literature available on natural fiber composites, particularly bagasse fiber composites efforts are put to understand the basic needs of the growing composite industry. The conclusions drawn from this is that, the success of combining vegetable natural fibers with polymer matrices results in the improvement of mechanical properties of the composites compared with the matrix materials. These fillers are cheap and nontoxic, can be obtained from renewable sources, and are easily recyclable. Moreover, despite their low strength, they can lead to composites with high specific strengths because of their low density.

Thus the priority of this work is to prepare Polymer Matrix Composites (PMCs) using bagasse fiber (waste from sugarcane industry) as reinforcement material and to study its weathering behaviour.

Structural polymer matrix composites (PMC's) are rarely subjected to hundreds of thermal cycles from extremely low temperatures to an elevated maximum service temperatures. However 20 years ago, composites were first considered for satellite applications in which dimensional stability was critical during exposure to alternating periods of +125 and -155 degree Celsius. When orbiting the earth. Significant micro-

cracking in any ply was considered detrimental to the coefficients of thermal expansions for that ply and consequently dimensional stability of the laminate. Recently a set of applications involving cryogenic and elevated temperature excursions has gained attention. A few studies specifically addressing the mechanical property changes and damage accumulation resulting from a combined cryogenic temperature and elevated temperature thermal cycle are available. Different loading conditions are probable in many of the applications where fiber reinforced polymers composites find use as potential and promising material. Damage and failure on a small scale in cryogenic liquid-storage systems is expected to grow and spread with freeze thaw cyclic and it may eventually cause bulk failure in structure that were not designed to account for micro-cracking and residual stresses induced by thermal shock and thermal cycling. The effect of varying loading rate on mechanical properties of fiber reinforced polymer composites has been in investigated and reported a variety of contradictory observations and conclusions. The polymer composites are characterized by a greater level of micro cracking and delamination at sub zero temperatures because of higher residual thermal stresses. The freeze thaw exposure can result in significant change in thermo mechanical response of polymer composite. Delamination and micro cracking are some of the most frequently observed damage phenomena that can develop in polymer composites when they are subjected to cryogenic conditioning. The use of polymer composites in safety critical applications leads to uneasiness since the mechanical response is not well established in freeze thaw condition in different load bearing conditions. The present work aims to study the role of interface in freeze thaw response at different loading rates and also by considering the weight fraction effect of constituent phases of glass epoxy composites.

CHAPTER 3

MATERIALS AND METHODS:

3.1 RAW MATERIALS:

Raw materials used in this experimental work are :

1. Natural fiber (Bagasse)
2. Epoxy resin
3. Hardener

3.1.1 BAGASSE FIBER

The sugar cane bagasse is a residue widely generated in high proportions in the agro-industry. It is a fibrous residue of cane stalks left over after the crushing and extraction of juice from the sugar cane. Bagasse is generally gray-yellow to pale green in colour. It is bulky and quite non uniform in particle size. The sugar cane residue bagasse is an under utilized, renewable agricultural material that consist of two distinct cellular constituents. The first is a thick walled, relatively long, fibrous fraction derived from the rind and fibro-vascular bundles dispersed through out the interior of the stalk. The second is a pith fraction derived from the thin walled cells of the ground tissue.

The main chemical constituents of bagasse are cellulose, hemicellulose and lignin. Hemicellulose and cellulose are present in the form of hollow cellulose in bagasse which contributes to about 70 % of the total chemical constituents present in bagasse. Another important chemical constituent present in bagasse is lignin. Lignin acts as a binder for the cellulose fibers and also behaves as an energy storage system.

Fig 3.1: BAGASSE FIBER

PREPARATION OF COMPOSITES

3.1.2 EPOXY RESIN

Softener (Araldite LY 556) made by CIBA GUGYE limited having the following outstanding properties has been used as the matrix material.

a. Excellent adhesion to different materials.

b. High resistance to chemical and atmospheric attack.

c. High dimensional stability.

d. Free from internal stresses.

e. Excellent mechanical and electrical properties.

f. Odorless, tasteless and completely nontoxic.

g. Negligible shrinkage.

3.1.3 HARDENER

In the present work hardener (HY951) is used. This has a viscosity of 10-20 MPa at 25ºc.

3.2 PREPARATION OF COMPOSITES:

The following procedure has been adopted for the preparation of the specimen.

3.2.1 Bagasse fiber preparation:-

Fresh bagasse fibers were collected after they were crushed for extracting juice by using a hand crushing machine. These fibers were then spread on a water proof sheet to reduce the moisture content. After approximately two weeks, the long bagasse fibers were shortened into a length of 10mm, breadth of 1mm and width of 1mm with a pair of scissors. Small size fibers were selected in order to design a composite with consistent properties. Due to the low moisture content of the bagasse samples, no fungi grew during the storage. The bagasse samples were then cleaned via pressurized water for about one hour. This procedure removes fine bagasse particles, sugar residues and organic materials from the samples. Then the fibers were dried with compressed air.

3.2.2 Composite preparation:-

A wooden mold of dimension (120x100x6) mm was used for casting the composite sheet. The first group of samples were manufactured with 5, 10, 20 % volume fraction of fibers**.** For different volume fraction of fibers, a calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed with gentle stirring to minimize air entrapment. For quick and easy removal of composite sheets, mold release sheet was put over the glass plate and a mold release spray was applied at the inner surface of the mold. After keeping the mold on a glass sheet a thin layer (≈ 2 mm thickness) of the mixture was poured. Then the required amount of fibers was distributed on the mixture. The remainder of the mixture was then

poured into the mold. Care was taken to avoid formation of air bubbles. Pressure was then applied from the top and the mold was allowed to cure at room temperature for 72 hrs. This procedure was adopted for preparation of 5, 10 and 20% fiber volume fractions of composites. After 72 hrs the samples were taken out of the mold, cut into different sizes and kept in air tight container for further experimentation.

3.3 EXPERIMENTAL PROCEDURE:

3.3.1 Liquid Nitrogen: How it is maintained at its low temperature

The liquid nitrogen we use in the experiments in the course of this project is maintained at 77K i.e. (-196 degree Celsius). A brief description of the process as how the temperature is maintained and the temperature is reached is explained below.

The liquid nitrogen production process consists of the following parts:

a) Air compressor: N2 is available in its molecular form from the atmosphere where it comprises (78%). Here the air is compressed to (8-10 kg/sq. m).

b) A compressed air filter bank : Here the air which is compressed is filtered to get 98% pure nitrogen gas. The filter bank has two stage filter which fulfills the nitrogen gas form oxygen, moisture and other gases.

c) A nitrogen gas generator : This generator consists of an He-compressor when this helium is released to low pressure just above the atmospheric pressure due to sudden pressure drop its temperature drops suddenly and this temperature drop is transferred to the Dewar in which the gaseous nitrogen is liquefied. As a result the process goes on and the temperature of the liquid nitrogen is maintained.

d) A cryorefrigerator : This is a setup so that we can work at this low temperature and perform the tests there. It consists of a blower which sprays the liquid N2 over a heater which heats the liquid to gaseous form but that too at a low temperature.

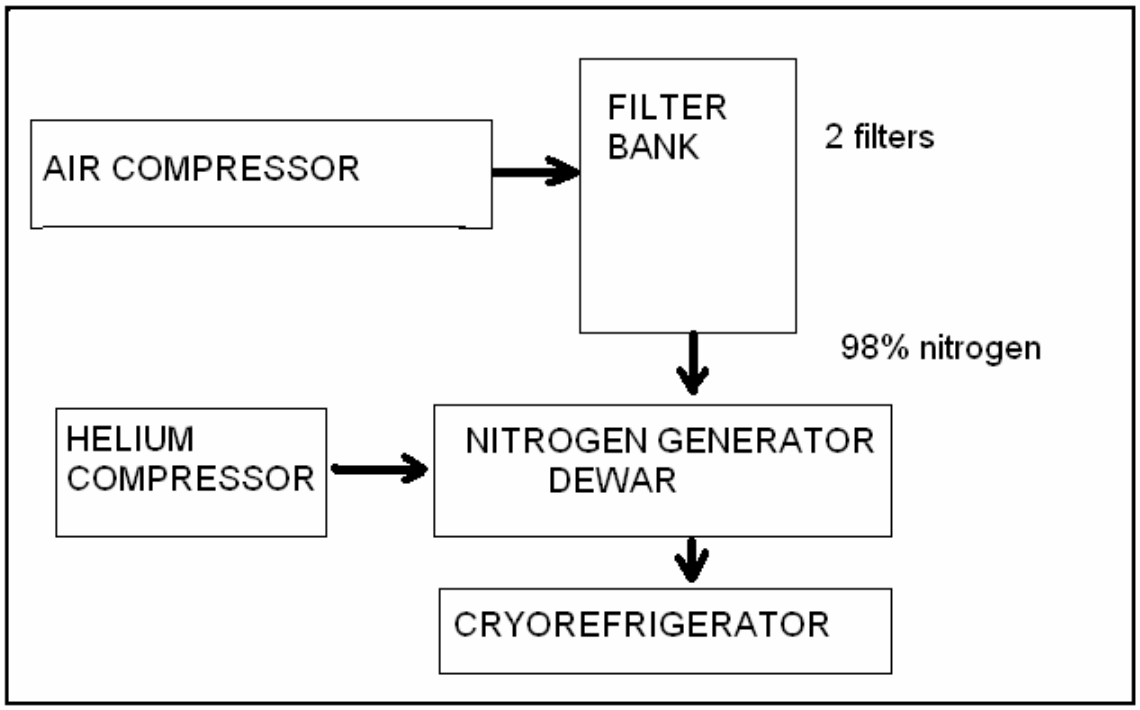


Fig 3.2: Schematic diagram for separation and production of liquid nitrogen

3.3.2 Cryogenic Treatment

The bagasse fiber/epoxy composite laminates were fabricated by wet lay-up method; The stability test was done for the composite laminates. Here the laminates were weighed and then heated in an oven at 50°C. The weight is intermittently checked till we get a stable weight, that is, with further heating there is no change in the weight of the composite. After the stability test the samples were allowed to come back to the ambient temperature. Then they were kept in the desiccators so that there is no further absorption of moisture. The samples to be cryogenically treated were exposed to a liquid nitrogen environment (77 K) for one hour. After the exposure, one batch of samples was taken out and kept at room temperature for one hour. Another batch of samples was tested in a 3-point bend test immediately after exposure to cryogenic temperature.

3.4 CHARACTERIZATION

3.4.1 Flexural strength measurement:-

The three point bend test were carried out for first batch of samples immediately after exposure to cryogenic temperature in an UTM 201 machine in accordance with ASTM D2344-84. The former samples after exposure to room temperature and the untreated as-cured samples were tested in short beam shear test at room temperature. All the specimens (composites) were of rectangular shape having length varied from 100-125 mm, breadth of 100-110 mm and thickness of 4-6 mm. A span of 60 mm was employed maintaining a cross head speed of 100mm/min. The flexural strength found out from the experiment are presented in table 4.1.

The flexural inter laminar shear strength (ILSS) of the composite which is the maximum shear stress that a material can withstand before it ruptures, was calculated using the equation

$$\sigma m = 3f/4bt$$

Where σm is the ILSS, f is the load, b is the width and t is the thickness of the specimen under test. The maximum tensile stress was found out form the equation.

$$\tau m = 3fl/2bt^2$$

Where τm is the maximum tensile stress and l is the gauge length.

CHAPTER 4

RESULTS AND DISCUSSION:

4.1 RESULTS AND DISCUSSION:

Figure 4.4 shows the effect of volume fraction of fibers on flexural strength values at cryogenic temperature, ambient temperature after exposure to cryogenic temperature.

The matrix acts as the load transfer medium between the fibers, hence the performance of the composite depends on the capability of the matrix to transfer load through the interface. It is clear from the figure that the composites with 20% volume fraction of fibers show higher strength than the others. This can be due to less effective bonding with the available polymer matrix per unit surface area of the fibers. And also the flexural strength of the composites at cryogenic temperature has higher values than that at ambient temperature after exposure to cryogenic temperature. The higher FS values for cryogenically conditioned samples are attributed to an enhanced mechanical keying factor by the generation of cryogenic compressive stresses which enhances the friction at the interface due to contraction of epoxy matrix at low temperature.

Figure 4.5 shows the effect of fiber treatment on flexural strength values at cryogenic temperature, ambient temperature after exposure to cryogenic temperature. It is observed that the composites with untreated fibers showed higher values at cryogenic temperature than that of the treated samples.

4.2 FRACTOGRAPHIC ANALYSIS

Figure 3.6 shows the fractured surfaces of 10, 15 and 20% volume fraction fiber composites respectively at cryogenic temperature. It is clear from the scanning micrographs that the improvement in the strength value after cryogenic conditioning is probably due to differential thermal contraction (Figure 4.6a) of the matrix during

sudden cooling which leads to the development of greater cryogenic compressive stresses and may increase the resistance to debonding and better adhesion by mechanical keying factor at the interface between fiber and the matrix.
The matrix contracts (Figure 4.6b) when the temperature decreases due to which internal stresses are generated in the matrix. Destruction of the matrix is induced when the thermal stress exceeds the strength of the resin.

It was observed that more matrix residue adhered (Figure 4.6c) to the fiber for cryogenically conditioned specimens. This may be due to increase in friction and adhesion between fiber-matrix depending on the compression by contraction of matrix as the polymer contracts more than the fiber.

Table-4.1

Flexural strength of 10, 15 and 20% volume fraction of composites at cryogenic and ambient temperature after exposure to cryogenic temperature.

Volume fraction of fibers in composites (%)	Flexural strength (MPa)	
	Cryogenic Temperature	Ambient Temperature
10	67.5	48.56
15	71.1	51.68
20	102.26	55.85

Table-4.2

Flexural strength of 20% treated and untreated fiber volume fraction of composites at cryogenic and ambient temperature after exposure to cryogenic temperature.

Types of fiber treatment (20% Volume fraction composites)	**Flexural strength (MPa)**	
	Cryogenic Temperature	**Ambient Temperature**
Untreated	102.26	55.85
Alkali	50.34	55.06
Acetone	36.84	34.05

Fig 4.1 Soxhlet extractor

Fig 4.2 Testing machine with the specimen in loading position

Fig 4.3 Different loading position of the specimen in the Testing Machine

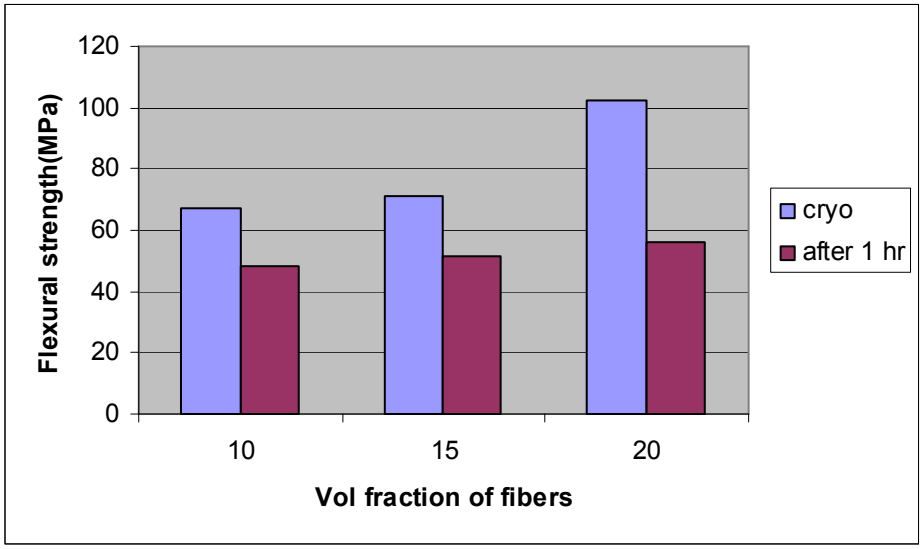


Fig 4.4 Variation of flexural strength values of 10, 15 and 20% volume fraction of composites at cryogenic and ambient temperature after exposure to cryogenic temperature.

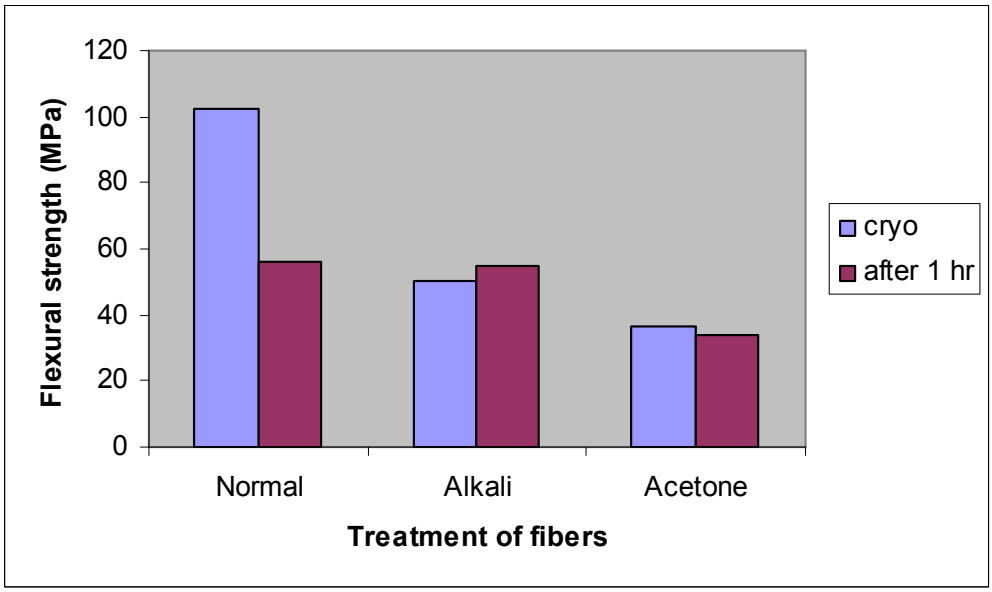
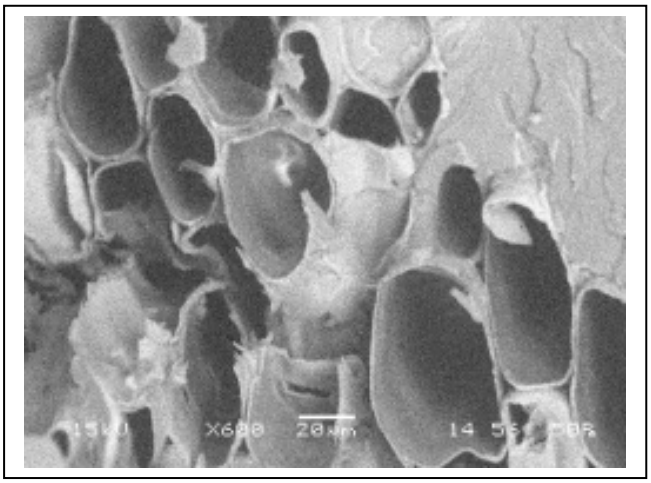
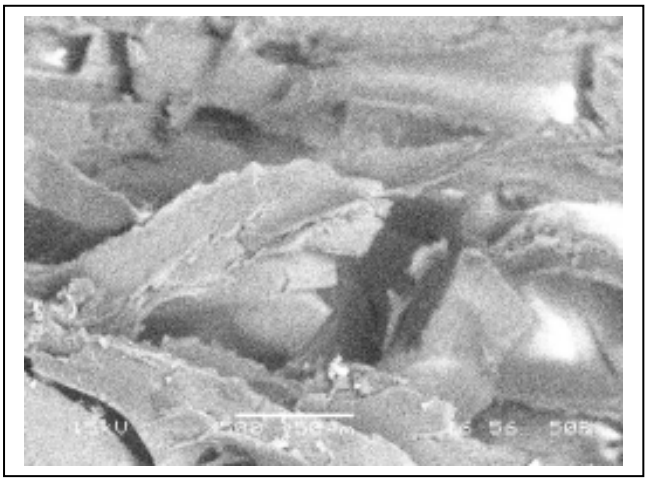


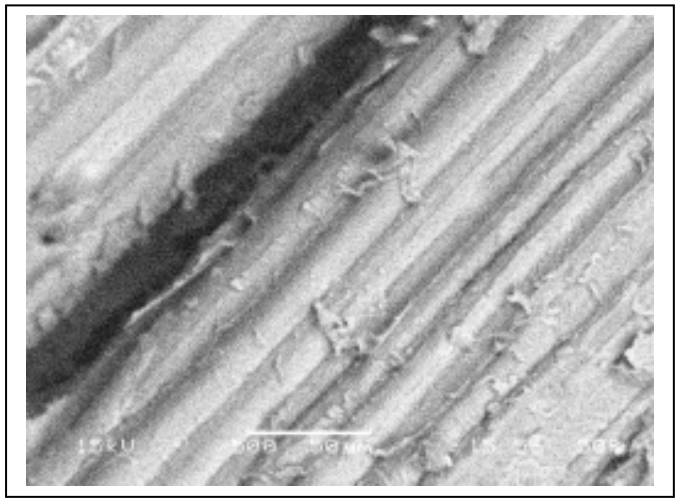
Fig 4.5 Variation of flexural strength values of 20% treated and untreated fiber volume fraction of composites at cryogenic and ambient temperature after exposure to cryogenic temperature.



(a)



(b)



(c)

Fig 4.6 Fracture surface of composites (10, 15 and 20% volume fraction of fibers) at cryogenic temperature

CHAPTER 5
CONCLUSION:

5.1 CONCLUSION:

The flexural strength of the composites at cryogenic temperature has higher values than that at ambient temperature after exposure to cryogenic temperature. The higher FS values for cryogenically conditioned samples are attributed to an enhanced mechanical keying factor by the generation of cryogenic compressive stresses which enhances the friction at the interface due to contraction of epoxy matrix at low temperature. It is observed that the composites with untreated fibers showed higher values at cryogenic temperature than that of the treated samples.

The improvement in the strength value after cryogenic conditioning is probably due to differential thermal contraction of the matrix during sudden cooling which leads to the development of greater cryogenic compressive stresses and may increase the resistance to debonding and better adhesion by mechanical keying factor at the interface between fiber and the matrix.

The matrix contracts when the temperature decreases due to which internal stresses are generated in the matrix. Destruction of the matrix is induced when the thermal stress exceeds the strength of the resin.

It was observed that more matrix residue adhered to the fiber for cryogenically conditioned specimens.

5.2 RECOMMENDATION FOR FURTHER RESEARCH :

----In this study fiber volume fraction of 20% has been used. This can be further increased to find out the optimum filler volume fraction.

----The fiber matrix bonding has been increased by treating the fibers with acetone, alkali.. Improvements in the process of bagasse cleaning and surface treatment could increase the performance of the composite and provide better competitiveness with respect to other materials in the same structural class.

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